Effect of helium ion implantation and annealing on the relaxation behavior of pseudomorphic Si$_{1-x}$Ge$_x$ buffer layers on Si (100) substrates

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The influence of He implantation and annealing on the relaxation of Si$_{0.7}$Ge$_{0.3}$ layers on Si (100) substrates is investigated. Proper choice of the implantation energy results in a narrow defect band ≈ 100 nm underneath the substrate/epilayer interface. During annealing at 700–1000 °C, He-filled bubbles are created, which act as sources for misfit dislocations. Efficient annihilation of the threading dislocations is theoretically predicted, if a certain He bubble density with respect to the buffer layer thickness is maintained. The variation of the implantation dose and the annealing conditions changes density and size of spherical He bubbles, resulting in characteristic differences of the dislocation structure. Si$_{1-x}$Ge$_x$ layers with Ge fractions up to 30 at. % relax the initial strain by 70% at an implantation dose of 2×10^{16} cm^{-2} and an annealing temperature as low as 850 °C. Simultaneously, a low threading dislocation density of 10^7 cm^{-2} is achieved. The strain relaxation mechanism in the presence of He filled bubbles is discussed. © *2002 American Institute of Physics.* [DOI: 10.1063/1.1504496]

I. INTRODUCTION

"Virtual" substrates in the form of strain relaxed and rather defect-free Si$_{1-x}$Ge$_x$ layers on Si (100) are the key to control the strain and, by that, the band gap and band offset of Si/SiGe layers grown onto such substrates. Following this concept, highly attractive heterostructures such as hetero-field-effect transistors (FETs) can be fabricated.[1–3] The promising application in high speed electronic devices based on Si technology has triggered many efforts to tackle the basic problem: Initially, Si$_{1-x}$Ge$_x$ layers deposited onto Si (100) substrates below the critical thickness are metastable, dislocation free, and tetragonally strained according to the lattice mismatch. On the one hand, these Si$_{1-x}$Ge$_x$ layers have to be transformed into the relaxed state without introducing a significant density of threading dislocations piercing the active device area. On the other hand, for many applications the Ge content has to be increased up to a value of 50% to provide optimum band gap and band offset values. Conventional relaxation methods like thermal annealing result in only a low degree of relaxation accompanied by a high density of detrimental threading dislocations, and are therefore not the method of choice.[4,5]

Many approaches have been developed to reduce the density of surface penetrating threading dislocations: By growth of several μm thick compositionally graded SiGe alloy layers[6,7] the threading dislocation density in SiGe (30%) buffers was reported to be as low as 10^4–10^7 cm^{-2}. Alternatively, thick Si$_{1-x}$Ge$_x$ layers were grown on very thin Si surface films of silicon-on-insulator wafers, which causes the threading dislocations to bend down toward the Si/SiO$_2$ interface, thus preventing a high threading dislocation density in the Si$_{1-x}$Ge$_x$ layer.[8] Thin strain relaxed SiGe buffer layers were formed by ion implantation of Ge into the thin Si layers of silicon-on-insulator substrates, where thermal annealing led to homogeneous Si$_{1-x}$Ge$_x$ layers directly on SiO$_2$.[9] In other approaches, SiGe buffer layers were deposited onto a low-temperature grown Si or SiGe buffer layer,[10,11] where the high intrinsic defect density injected into the low temperature layer enhances the nucleation of strain-relieving misfit dislocations and reduces the threading dislocation density considerably. However, successful results were only obtained for rather low Ge concentrations (<20%). A comparison of device properties of modulation doped quantum wells and FETs grown onto different types of SiGe buffers shows thick graded buffers to be better than low-temperature buffers, both of them being superior to constant composition buffers.[12]

Recently, high degrees of relaxation and low threading dislocation densities were achieved by H and He implantation and subsequent annealing of thin Si$_{1-x}$Ge$_x$ layers on Si (100) substrates.[13–15] The main advantage of this technique is that thin, strain-relaxed SiGe layers, which are produced by use of overpressurized H or He bubbles acting as internal dislocation sources, can be processed by simple, standard

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TABLE I. Summary of He implantation doses, annealing temperatures and times, and quantitative results of the structural analyses. All samples consist of a 100 nm thick $Si_{0.7}Ge_{0.3}$ layer grown onto Si (100) substrate.

Implantation dose (10^{16} cm^{-2})	Annealing temperature/time (°C)/(s)	Degree of relaxation (%)	Diameter of He bubbles (nm)	Mean distance of He bubbles (nm)	Threading dislocation density (10^7 cm^{-2})
0	750/600	7	...	...	...
	800/600	14	...	...	...
	850/600	34	...	...	...
	1000/30	34	...	...	...
1.2	850/600	70	15.1±4.9	49.5	45±1.8
2.0	as implanted	...	no bubbles observed	...	no dislocations observed
	750/600	66	8.59±4.59	9.3	3.7±1.8
	800/600	68	10.78±4.06	12.6	4.4±1.6
	850/600	68	15.39±4.89	18.3	1.62±0.8
	1000/30		22.99±6.96	24.1	102±39
2.8	850/600	83	15.92±5.61	13.8	181±1
3.0	850/600	80	13.4±3.63	13.7	7900±140
	1000/30			38.5	

technology steps. Depending on the implantation conditions, various types of He bubbles connected with different dislocation reactions were observed.[16] The interaction between bubbles and dislocations require the bubble layer to be separated from the interfacial region, i.e., best results are achieved if the bubbles are located below the interface.[15] Whereas H implantation has been found to be suitable for Ge concentration of up to about 20% only,[13] He implantation led to a successful relaxation of $Si_{1-x}Ge_x$ layers with higher Ge concentration of 30%.

In our article we investigate the relaxation behavior of $Si_{1-x}Ge_x$ layers on Si (100) substrates in dependence on He implantation and annealing conditions. The relaxation mechanism and He bubble formation are discussed on the basis of experimental results of structural characterizations by transmission electron microscopy (TEM).

II. EXPERIMENT

Metastable $Si_{1-x}Ge_x$ layers with Ge fractions of 30 at. % and a thickness of 100 nm, slightly below the critical thickness for pseudomorphic layer growth, were grown by molecular beam epitaxy on Si (100) standard wafers. The layers were fully strained according to the mismatch of −1.2% as determined by x-ray diffraction and ion channeling.[14] The heterostructures were implanted with He$^+$ ions with energies of 18 keV and doses in the range of 1.0×10^{16}–3.0×10^{16} cm^{-2} at room temperature with an implantation rate of 8×10^{12} cm^{-2} s^{-1}. The implantation energy was chosen such that a defect region at the end-of-range formed about 100 nm below the SiGe/Si interface.

After ion implantation the samples were annealed at 750–850 °C for 600 s or—in high-temperature samples—at 1000 °C for 30 s in a quartz lamp furnace under Ar atmosphere. For comparison, identical samples were annealed without ion implantation. The implantation and annealing conditions are summarized in Table I. The amount of strain relaxation was determined by ion channeling angular scans

along a (100) plane through an inclined [110] direction and by x-ray diffraction.[14] The structural properties were investigated by use of a JEOL 4000FX TEM operated at 400 kV. Samples were prepared in cross section and in plan view by conventional techniques including Ar ion milling.

III. RESULTS

The effect of He implantation and subsequent annealing at 850 °C on the formation of He bubbles[17] in SiGe/Si heterostructures is shown in the TEM bright field images in Fig. 1. All images are taken with kinematical imaging conditions in underfocus, which causes a bright contrast of the bubbles surrounded by dark Fresnel contours. Strain fields, e.g., of dislocations appear as blurred dark contrast under these imaging conditions. The cross section viewgraph in Fig. 1(a) reveals a well defined layer of spherical bubbles in a sample
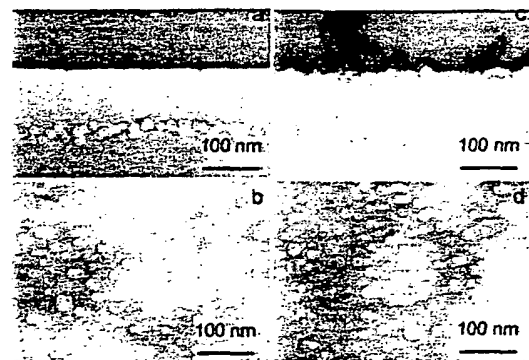

FIG. 1. The bright field electron micrographs show the $Si_{0.7}Ge_{0.3}$/Si(100) heterostructures in cross section and plan-view. Upon He implantation with 2×10^{16} cm^{-2} and annealing at 850 °C (a) and (b) a well defined bubble layer of laterally homogeneously distributed bubbles is observed. Implantation with 2.8×10^{16} cm^{-2} and annealing at 850 °C (c) and (d) results in a broadening of the bubble layer and the formation of bubbles within the interface.
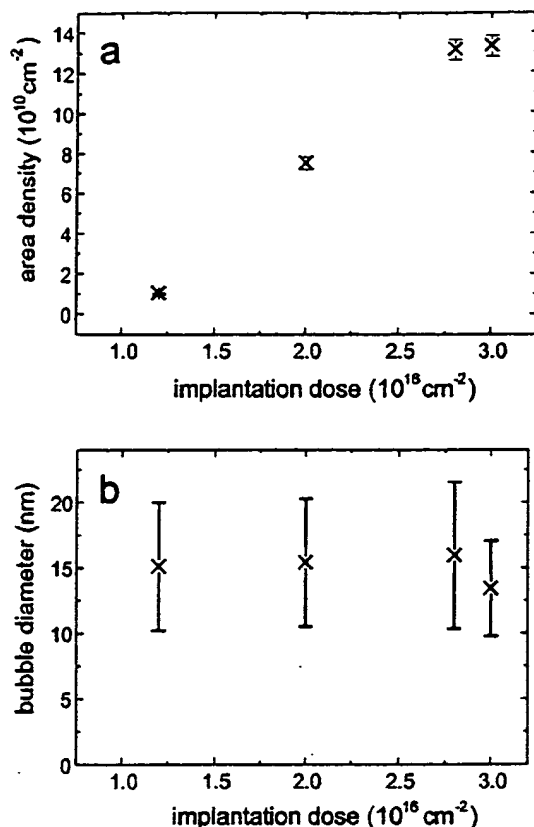
FIG. 2. The area density (a) and bubble diameter (b) are plotted vs the implantation dose. The annealing temperature of 850 °C is the same for all samples.

FIG. 3. Degree of relaxation vs the He implantation dose for the different annealing temperatures indicated. Samples at 0 cm^{-2} were not implanted but annealed only.

implanted with 2×10^{16} cm^{-2} He. According to the ion energy chosen, the layer is located about 100 nm below the SiGe/Si interface, i.e., within the Si substrate. A homogeneous lateral distribution of the bubbles is deduced from the corresponding plan-view image shown in Fig. 1(b). The shape of the bubbles is almost spherical with diameters ranging from 5 to 25 nm. Applying a higher implantation dose of 2.8×10^{16} cm^{-2} results in a considerably broader bubble layer as depicted in the cross sectional viewgraph [Fig. 1(c)]. Furthermore, large bubbles are located within the interface of the SiGe/Si heterostructure. Dark contrast within the SiGe epilayer is caused by threading dislocations. In plan view, again, a homogeneous distribution of the bubbles is detected. However, the density of the bubbles is considerably higher in Fig. 1(d) compared to the lower dose [Fig. 1(b)] imaged with the same magnification. The size of the spherical bubbles is about the same. Dynamical two beam imaging conditions were applied to investigate strain fields. It has to be emphasized that the bubbles do not show any strain contrast, and are therefore surrounded by an undistorted Si matrix. Few dislocations were found to be attached to some of the bubbles.

Quantitative assessment of the bubble density and size is shown in Fig. 2 for samples subjected to different implantation doses, while the annealing temperature (850 °C) and time (10 min) were the same for all cases. The area density of 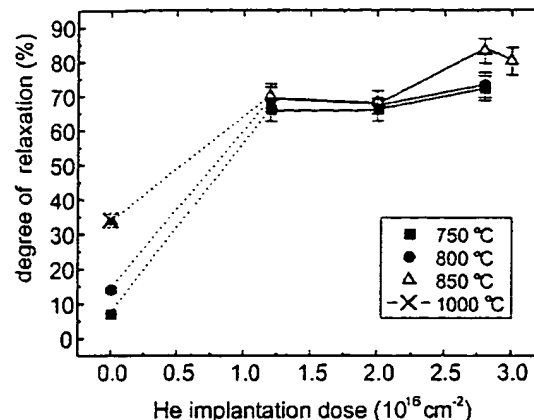the bubbles determined form plan-view electron micro-graphs is plotted in Fig. 2(a), which allows the comparison between samples with different, partly inhomogeneous depth distributions of the bubble layer [see e.g., Figs. 1(a) and 1(b)]. Clearly, an increase of the bubble density is observed for increasing implantation dose. The mean lateral distance of the He bubbles determined form the area density is given in Table I. These values reflect the "real" distance between precipitates, if the bubble layer is thin, i.e., at implantation doses $\leq 2 \times 10^{16}$ cm^{-2} and annealing temperatures ≤ 850 °C. As the bubble layer becomes thicker, the distance between bubbles is underestimated. The mean bubble diameter determined from measurements on about 200 bubbles in each sample remains constant with increasing He implantation dose [Fig. 2(b)]. The size of the error bars reflects the considerable scatter of bubble diameters within individual samples.

He implantation has a dramatic effect on the degree of relaxation of the SiGe layers grown on Si substrates. Figure 3 (and Table I) shows the degree of relaxation for samples subjected to different He implantation doses and annealing conditions. The values determined ·by Rutherford backscattering spectrometry from the Ge backscattered signal in angular scans are identical within the margin of errors with those measured by x-ray diffraction.[14] For comparison, data obtained by annealing without prior He implantation are included in the figure. Here, the maximum degree of relaxation of 35% is obtained for annealing temperatures larger than 800 °C. Considerably higher values are observed in He implanted and annealed samples. For all implantation doses and annealing temperatures applied, the degree of relaxation remains approximately constant at about 70%, except for the samples implanted at high doses and annealed at 850 °C, where even higher values of up to 83% are observed.

These high degrees of relaxation are accompanied by a high density of misfit dislocations located within the interface between the SiGe epilayer and the Si substrate. In all cases a regular dislocation network is revealed, the plan-view image in Fig. 4 being an example, where dislocations show bright contrast according to the weak beam dark field imaging conditions chosen. In this particular sample implanted
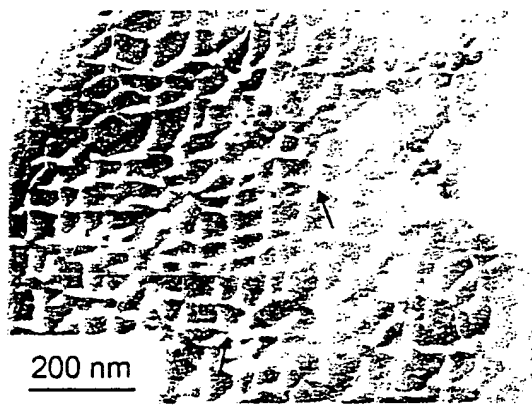
FIG. 4. Weak beam dark field electron micrograph displaying the misfit dislocation network. Dislocations show white contrast. Frequently, He bubbles can be seen to decorate the nodes of the network (arrows).

with 2×10^{16} cm^{-2} He and annealed at 1000 °C, a large number of bubbles is located within the interface, predominantly attached to the nodes of the network (see arrows in Fig. 4). Detailed contrast analyses reveal that the network consists mainly of 60° dislocations. In addition, some 90° dislocation are formed by reaction of two 60° dislocations with different Burgers vectors.

From a device point of view not only a high degree of relaxation is important but also low threading dislocation densities. The threading dislocation density was determined from plan-view samples where the Si substrate was removed. The result of counting the number of dislocations within a defined area of samples subjected to different He implantation doses and annealing temperatures is presented in Fig. 5. The threading dislocation density obtained after annealing at 1000 °C or at high implantation doses is always larger than 10^9 cm^{-2}, which is unacceptably high for any device application. A minimum threading dislocation density of about 10^7 cm^{-2} is observed for a He implantation dose of 2.0 $\times 10^{16}$ cm^{-2} and an annealing temperature of 850 °C.

In contrast to annealed samples, the as-implanted sample implanted with 2×10^{16} cm^{-2} does not reveal any structural defects, neither dislocations nor He bubbles.
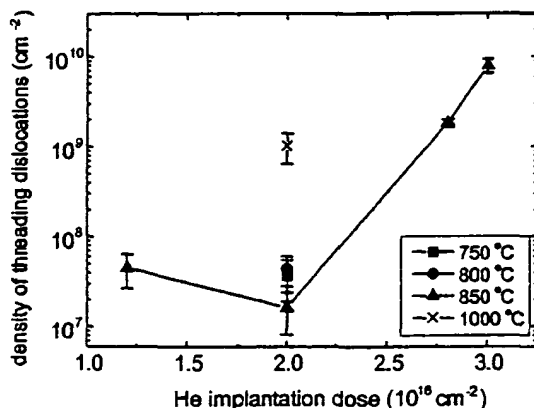

FIG. 5. Threading dislocation density plotted vs the He implantation dose for the different annealing temperatures indicated.

IV. DISCUSSION

Two aspects of the effect of He implantation and annealing on the relaxation of SiGe/Si heterostructures will be discussed below. First, we will address the formation of He bubbles within the Si substrate. Second, we will focus on the relaxation mechanism of the SiGe layers in the presence of the He bubbles acting as internal dislocation sources.

The formation of He bubbles in Si is a complex phenomenon, since several processes depending on annealing time, temperature, and implantation parameters are involved, like nucleation, decomposition, and He desorption. According to Fichtner *et al.*[18] three regimes of different nucleation and growth phenomena can be distinguished: At low He concentration ($< 1 \times 10^{16}$ cm^{-2}) He$_n$V$_m$ clusters of n He atoms and m vacancies (V) are formed, which dissociate at low annealing temperature (<400 °C).[19] At high He concentration ($>2 \times 10^{16}$ cm^{-2}), bubbles are already formed during implantation and follow a coarsening process during annealing.[20] Finally, in the intermediate He concentration regime the microstructure is strongly dependent on the annealing temperature, the as-implanted sample being free of any extended defects and He bubbles. Annealing at low temperatures (≈ 300 °C) results in platelike He precipitates, which are heavily overpressurized as indicated by the surrounding strain fields.[21] At this stage the formation of dislocation loops surrounding the platelets was observed. Annealing at higher temperatures (600 °C) results in a decomposition of the platelets into spherical bubbles, which are still surrounded by strain fields owing to the overpressurized state of the bubbles. Only upon high temperature anneals (>700 °C) is the pressure within the bubbles released by desorption of He.[20]

Clearly, at low doses ($\leq 2 \times 10^{16}$ cm^{-2}) our case is characterized by "intermediate He concentrations," since no bubble-like structures can be detected in the as-implanted sample. The independence of the bubble diameter of implantation dose (Fig. 2) indicates coarsening of the bubbles by Ostwald ripening during annealing, according to theoretical considerations[22] and experimental studies on He bubbles in metals.[23] Obviously, the bubble structures (Fig. 1) formed during annealing can be considered as a late state of their evolution, since no strain fields can be observed. Therefore, part of the He seems to be released by annealing. However, according to literature, in the early stages overpressurized bubbles or platelets are formed, which act as dislocation sources. Indeed, we find some dislocations within the layer and, more importantly, a considerably higher density of misfit dislocations within the SiGe/Si interface compared to unimplanted and annealed only samples.

The role of He bubbles as internal dislocation sources is the key to producing relaxed SiGe layers on Si substrates with only few threading dislocations. Conventional relaxation of heterostructures through annealing involves the heterogeneous generation of dislocations at internal dislocation sources such as grown-in dislocations or at the surface of the epilayer, which glide into the interface and thereby relax the layer.[24] If this procedure is imposed on our samples, the maximum degree of relaxation achievable is about 35% only
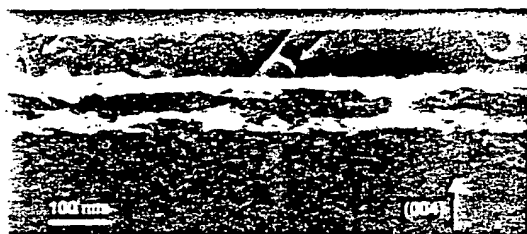
FIG. 6. Weak beam dark field electron micrograph in cross section of a sample implanted with 2×10^{16} cm^{-2} and annealed at 1000 °C. The bubble layer and the interface show blurred white contrast according to the strain fields of dislocations. Clearly, two dislocation loops can be seen to be generated within the sample, whereas a half loop is attached to the surface.

(see Fig. 3 and Table 1). Introducing He bubbles into the Si substrate improves the degree of relaxation by a factor of 2. Evidence for dislocation loop generation from internal sources is given in Fig. 6. The weak beam dark field image shows two dislocation loops extending from the SiGe/Si interface into the epilayer, whereas one dislocation loop is evidently formed at the surface. Thus, two relaxation mechanisms are active in this particular sample, implanted with 2×10^{16} cm^{-2} and annealed at a very high temperature of 1000 °C.

Recently, the strain relaxation by H and He implantation of SiGe/Si heterostructures was described in a theoretical model[25] which suggests that the strain relaxation was initiated by dislocation loops punched out by the He filled overpressurized bubbles within the Si substrate. These loops glide to the interface. When a loop comes into contact with the SiGe layer it experiences an asymmetric force. As a consequence, one side of the loop is pinned at the interface where it forms a strain relieving misfit segment. The other side is driven by the mismatch stress to the surface where an atomic step is generated. The accompanied threading dislocations from different loops annihilate efficiently by a combined climb–glide mechanism. Obviously the density of dislocation sources, i.e., He bubbles, has to be sufficiently high to allow generation of a large number of misfit dislocations to relax the SiGe layer. In addition, the distance between threading dislocations has to be smaller than the annihilation radius of two dislocations with opposite Burgers vectors. Trinkaus et al.[25] quantified two conditions necessary for effective relaxation and complete annihilation of threading dislocations: $\delta \leqslant 2.5d$ and $\delta \leqslant h$, where δ is the distance between precipitates, d the bubble diameter, and h the thickness of the epilayer.

These conditions appear to be well fulfilled for all our samples: Taking the implantation series (see Table I) as an example, δ varies between 13 and 49 nm, which is within the margin of errors smaller than or equal to 2.5 $d = (38 \pm 12)$ nm. Thus, even samples with high He implantation doses and/or high annealing temperatures seem to fulfill the conditions required by the model. However, in these cases the annihilation of the threading dislocations does not work efficiently. This may indicate that the conventional relaxation mechanism still operates (see Fig. 6), introducing threading dislocations, which cannot effectively annihilate. In addition, these samples contain a considerable density of bubbles

within the interface. These react with the dislocations[26] and therefore impede motion of the threading dislocations necessary for annihilation.

It is important to point out that the initial overpressure within the bubbles is not known to date, which prevents quantitative evaluation of their strength as dislocation sources. Since the bubbles observed in the present study do not exhibit strain fields, dislocation formation must occur in an earlier stage of the bubble evolution, where the stress imposed by an overpressurized bubble is larger than the critical shear stress for dislocation formation. Indeed, dislocation loops have been reported to form already at temperatures as low as 350 °C, where the bubbles still have their platelet shape.[18] Therefore, assuming the dislocation generation in the "platelet stage," the density of dislocation sources can be considerably lower as deduced from the density of spherical bubbles, since the mean distance between platelets is larger than the spacing between bubbles observed at a later stage. In consequence, an effective dislocation source density has to be considered in the model[25] to quantitatively describe the relaxation mechanism. Qualitatively, our experimental observations of the bubble structure and of the types of dislocations are in agreement with the model.

V. CONCLUSION

In conclusion, a high degree of relaxation of thin SiGe layers grown on Si (100) substrates can be achieved by He implantation and subsequent annealing at relatively moderate annealing temperatures. The dislocation generation necessary for relaxation of the $Si_{0.7}Ge_{0.3}$ layer is induced by the presence of overpressurized He filled precipitates. At a dose of 2.0×10^{16} cm^{-2} and an annealing temperature of 850 °C a threading dislocation density as low as 10^7 cm^{-2} is obtained. Therefore, He implantation and subsequent annealing are promising techniques to achieve good quality strain-relaxed SiGe grown on Si (100), even for layers with Ge concentrations of as high as 30%. Such wafers are highly attractive as virtual substrates for Si/SiGe based high speed/high performance devices in Si technology. The advantages compared to thick strain relaxed buffer layers are quite obvious: The small thickness of the SiGe film poses no problems in technology and, especially important, it minimizes the problem of low thermal conductivity of SiGe alloys.

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